PATENT
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants: Donald B. Appleby et al : Group Art Unit: 1211

Serial No.: 08/360,184 : Examiner: E. White

Filed: December 20, 1994 :

For: **Polyol Polyester Synthesis**

DECLARATION UNDER 37 C.F.R. §1.608 OF GENE P. HAWKINS

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, GENE P. HAWKINS, declare that:

1. I was employed by the assignee of the present application, The Procter &

Gamble Company, from 1964 to 1998.

2. I received an Associate Degree in Chemical Engineering from the University of

Cincinnati in 1969.

3. From 1987 to 1990 I worked as a technician on the sucrose polyester synthesis

project in which sucrose and fatty acid methyl ester were reacted to form sucrose fatty acid

esters. I was responsible for analyzing samples of reactor outlet streams from pilot plant continuous sucrose polyester production processes according to established procedures.

4. I participated in the pilot plant continuous sucrose polyester production process designated P90117 which was conducted from January 18, 1989, to January 27, 1989. The P90117 pilot plant continuous sucrose polyester production process, including operation of the continuous sucrose polyester synthesis equipment, sample collection and data preparation, was performed under the direction and control of Mr. Scott Pearson.

5. During the P90117 pilot plant continuous sucrose polyester production process, I determined the average degree of sucrose esterification (based on mole fractions and referred to as the I-bar value) and the area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in samples taken from the recirculation loops of the reactors according to established procedures described below. I accurately recorded the determined average degree of sucrose esterification (I-bar) and area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in the samples on data sheets which were maintained in the pilot plant for the purpose of recording such data. The data which I accurately recorded included the date and time at which a sample was taken, the reactor recirculation loop from which the sample was taken, and the determined average degree of sucrose esterification (I-bar) and area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in the sample. I also initialed the entry for each sample which I analyzed.

6.   I have examined Exhibit 8, and I confirm that Exhibit 8 comprises accurate copies of the data sheets from the P90117 pilot plant continuous sucrose polyester process into which I accurately entered the date and time at which a sample was taken, the reactor recirculation 100p from which the sample was taken, and the determined average degree of sucrose esterification (I-bar) and area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in the sample. The entries in Exhibit 8 under the heading "SFC" represent area percentages determined using supercritical fluid chromatography according to the established procedures described in the following ¶7. The entries in Exhibit 8 under the heading "HPLC" represent area percentages determined using high performance liquid chromatography according to the established procedures described in the following ¶8. Generally, SFC was used during the P90117 pilot plant continuous process to determine area percentages of sucrose esters in samples from reactors R600, R601 and R602 while HPLC was used during the P90117 pilot plant continuous process to determine area percentages of sucrose esters in samples from reactors R603, R604, R605 and R606. The entries bearing my initial (H) indicate those samples which I analyzed as described herein and for which I recorded the data as described herein.

7.   The established procedure for determining the area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester, respectively, in the sucrose ester, in a sample by supercritical fluid chromatography (SFC) was as follows: Each reactor sample was silylated to derivatize any unreacted hydroxyl groups and the silylated sample was injected into a supercritical fluid chromatograph. In the chromatograph, the esters were separated by degree of esterification on a capillary column and detected by a flame

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ionization detector. The detector output comprised a graph containing a series of peaks representing, respectively, the distribution of individual sucrose esters, on an area basis, in the sample. The area percentages of the individual sucrose esters relative to the total sucrose ester content in a reactor sample were determined using the equation:

$$\% \, SE_n = \frac{Area \, SE_n}{\sum Area \, SE} \times 100$$

wherein n indicates the number of ester groups on a sucrose ester, Area SE_n is the area under the peak corresponding to the "n" ester and $\sum$ Area SE is the sum of all of the areas under the ester peaks.

8.　The established procedure for determining the respective area percentages of the sucrose esters in a sample by high performance liquid chromatography (HPLC) was as follows: Each reactor sample was dissolved in hexane, and the resulting solution was filtered and then injected into a high performance liquid chromatograph equipped with a silica column. The mobile phase for the chromatography was a methyl-t-butyl/hexane step gradient system. The respective sucrose ester area percentage in the sucrose ester sample was calculated by the chromatograph integrator based on output from the chromatograph light-scattering mass detector.

9.　The I-bar was calculated on a mole fraction basis from the area percentages of monoester, diester, triester, tetraester, pentaester, hexaester, heptaester and octaester determined by chromatography, assuming the area percentages of the respective sucrose esters are substantially equivalent to weight percentages, according to the equation:

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$$I\text{-Bar} = \frac{\sum_{n=1}^{8} n \times \dfrac{\text{Area \% n ester}}{\text{Mol. Wt. n ester}}}{\sum_{n=1}^{8} \dfrac{\text{Area \% n ester}}{\text{Mol. Wt. n ester}}}$$

wherein "Mol. Wt." is the molecular weight of the respective ester.

10.   During the sucrose polyester synthesis project, I was also responsible for conducting sucrose polyester laboratory experiments wherein sucrose and fatty acid methyl ester were reacted to form sucrose fatty acid polyesters (sucrose polyesters) according to predetermined test plans and analyzing the results (or having the results analyzed under my direction and control) according to established procedures under the direction and control of Mr. Scott Pearson, including those described ¶¶ 11-12 herein. For each experiment which I conducted and which is described in this Declaration, I accurately recorded, on or about the day of the experiment, the general nature of the experiment, including pertinent reaction parameters, the results of the experiment, the date of the experiment and my signature in a Laboratory Notebook assigned to me for such a purpose.

11.   I have reviewed Exhibit 27 and confirm that it comprises accurate copies of pages 6-9 of Laboratory Notebook SI 6051 on which I accurately recorded a series of experiments I conducted on or about December 11, 1989 through January 8, 1990 and which I signed and accurately dated upon the completion of the procedures described on each page respectively. Laboratory Notebook SI 6051 was in my possession and control for at least between December 1989 and February 1990.

12.   As set forth on page 6 of Exhibit 27, the experiment described therein was intended to investigate, inter alia, the effect of using higher pressures in a packed column reactor in later stages of the sucrose polyester reaction. As indicated in the Table set forth on pages 8-9 of Exhibit 27, reaction pressures of from 15 mm Hg to 100 mm Hg at the top of the packed column were investigated.

13.   I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By: _GENE P. HAWKINS_

Date: _3-30-99_

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